5-1-1941

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Submitted to the Department of Mineral Dressing
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

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BUTTE, MONTANA
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A STUDY OF THE CONCENTRATION OF A MANGANESE-IRON ORE

The ore investigated in this thesis is principally a low grade manganese-iron ore coming from the Ennis district, Montana.

The purpose of the investigation was to determine the most effective means of concentrating the manganese in the ore. The first study was that of identification of the minerals present. This showed the presence of oxides of manganese and iron, and carbonates of calcium and manganese. Two methods of investigation were planned for the work: First, it was determined whether the ore could be concentrated effectively by gravity concentration methods. These tests failed to produce a concentrate of sufficiently high manganese content, although they did separate the lighter carbonates from the heavier manganese and iron minerals with a recovery of not less than 95 per cent of the total manganese present. Microscopic work and petrographic analysis were then neccessary to determine the association of hematite with the manganese minerals. These investigations failed to prove any definite replacement theories and it was concluded that the hematite should be liberated in the finer sizes. Roasting under a reducing atmosphere to convert the hematite to magnetite was then investigated. A Davis Magnetic Tube Concentrator was used to effect a separation of the magnetite from the manganese minerals.

The procedure followed in the various tests was similar to that as outlined in the United States Bureau of Mines publication, Report of Investigations 3328.

Geology of Ore Deposit[1]

Manganese ore bodies occur a short distance south of Cherry Creek about a mile west of Madison Valley. Cherry Creek is about 12 miles south of Wigwam Creek and 42 miles south of Norris. The smooth surface is modified by low rounded, barren hills; the relief being from 100 to 200 or 300 feet.

The manganese bearing area is underlain by limestone resembling those of the Cambrian deposits. The limestone is discolored by oxides of iron. Zones showing brownish-red shades range from 20 to 500 feet in width, for about 3 miles along a course of about N20°E. Manganese oxide minerals occur rather sparingly as streaks and small masses along the cleavage lines. An analysis shows this rock to contain 0.82 per cent manganese, 9.48 per cent iron, and 4.06 per cent insoluble matter.

No bedding is seen at Cherry Creek. The smooth surface mentioned passes beneath rhyolite lava in places which is a continuation of the Tertiary surface.

About 10 pits, shafts, and tunnels, none of which reach a depth greater than 40 feet, have been dug in this district. The ore bodies are irregular, but most of them approach flat lenselike or pipelike forms. Most of the ore bodies show more or less distinctly a zoned or layered structure, and the richest ore is generally found in a more or less distinct middle streak or core.

Psilomelane forms the bulk of the ore. It is generally massive but in places exists as botryoidal crusts that surround cores of softer oxides.

It is believed that the areas of discolored limestone in this territory are worth careful prospecting in the expectation of finding other ore bodies.

1 - U. S. Geological Survey, Bulletin 690, Pp. 131

Crushing and Sampling

Six 50 pound samples of ore were acquired from the Ennis district, through the Mineral Survey of the Montana Bureau of Mines. A portion of each of these samples was cut out by means of a Jones' splitter. Analyses are presented in Table I.

Table 1

Sample	Location	% Fe	% Mn	% Insol.	oz./ton	
					Au	Ag
1.	Portal of west adit	20.2	3.2			
2.	Upper adit dump		3.7		.005	.2
3.	Lower adit dump		1.5			
4.	Shaft dump	33.6	5.5	7.5		
5.	Hand picked sample	16.6	27.3	3.1		
6.	Sorted ore at shaft		3.7			

These analysis showed that only the No. 5 hand picked sample contained sufficient manganese to warrant further investigation. This sample was crushed directly through the rolls to -8 mesh. Any oversize from the rolls was crushed on the bucking board to pass 8 mesh.

Screen Analysis

A sample sufficient for screen analysis was cut from No. 5 sample. The sample was dry-screened on the Tyler Ro-Tap machine to 200 mesh. No weight was taken of the head sample so that dust losses were distributed throughout the different products. The results of the screen analysis are given in Table II

Table II

Screen Analysis of Head Sample

Mesh	Weight (grs.)	Weight %	Cum. Weight %
-8-10	128	16.5	16.5
14	128	16.8	33.3
20	125	16.1	49.4
28	72	9.3	58.7
35	69	8.9	67.6
48	54	7.0	74.6
65	41	5.3	79.9
100	40	5.1	85.0
200	52	6.8	91.8
-200	63	8.2	100.0
	774	100.0	

Microscopic Identification of Minerals

A microscopic examination of the ore was first made to determine the minerals present. For a study of opaque minerals, the specimens were mounted in bakelite briquettes and examined under reflected light.

Six specimens were taken of both No. 4 and 5 samples. The specimens were prepared and mounted by the following procedure: The samples were first ground with 600 carborundum on a glass plate until a surface was obtained free from scratches and pits. The specimen was placed on the plunger of the briquetting machine and a calculated quantity of bakelite added so as to give a briquette of approximately 15 cc. volume. A pressure of 6000 pounds per square inch was applied to the plunger, and by means of an electrical heating unit, the mold was heated to approximately 55° C. When the temperature reached 38°, additional pressure was applied until there was a total of 10,000 pounds per square inch. The heat was turned off when the temperature reached 55°. When the pressure became constant a short time was allowed to elapse (1 minute was sufficient) and the briquette was then removed from the machine. The briquette was trimmed and given a preliminary polish with 600 carborundum.

The briquette was then given a more complete finish on an automatic polishing machine, using copper and lead laps. The lubricant for each lap was a mixture of three parts of kerosene and one part of mineral oil. The amount of lubricant must be closely controlled, as too much or too little causes the specimen to pit. The grinding media for the copper and lead laps was minus 10 and minus 5 micron alundum, respectively. The specimens were ground on the copper lap to a fine matt surface. Machine

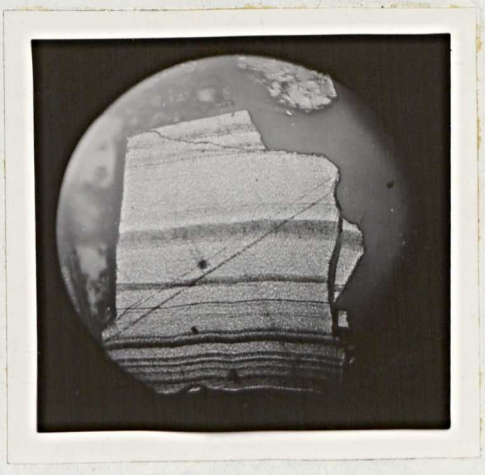
polishing was completed on the lead lap. A final polish was effected by hand on a tin oxide-impregnated chamois lap.

Examination of the specimens was carried out with a Bausche Lomb microscope using an oil immersion lens. The minerals and means of identification were as follows:

Hausmanite---Bluish white; harder than needle; showing strong anisotropism, polysynthetic twinning, and a red internal reflection.
Psilomelane--Greyish brown, showing anisotropism and a radial structure.
Hematite-----The harder residual grains-Light grey and anisotropicm.
Calcite------Effervesence with HCl.
Rhodocrosite or Dolomite questionable.

Plate I

300 X
(a)



300 X
(b)

(a) Photomicrograph showing the darker manganese minerals and the residual grains of hematite.
(b) Banded psilomelane taken from a briquette of minus 8 plus 10 mesh material.

Spectrographic Analysis

Four specimens were taken from No. 4 and 5 samples and prepared for spectrographic analysis. These samples were not briquetted. All polishing was accomplished by hand with 600 carborundum and -10 alundum on glass plates, and finished on the tin oxide-impregnated chamois lap.

A sample was chipped out of each of these four specimens and ground in an agate mortar for spectrographic analysis. Plate II is a photograph of the spectrum. The negative is used in determining the minerals present. Table III shows the results of the analysis.

Plate II

Table III

	(a) Hausmanite	(c) Psilomelane	(d) Carbonate	(e) Mn Mineral
K	tr	--	--	--
Cu	tr	--	tr	tr
Ag	--	--	--	--
Au	--	--	--	--
Mg	--	--	pres.	--
Si	pres.	pres.	3	2
Co	'	'	3	1
Zn	--	--	--	--
Sr	--	--	--	--
Ba	½	½	¼	1/8
B	--	--	--	--
Al	'	½	½	½
Ti	--	--	--	--
Sn	From polishing machine			
Pb	tr	tr	tr	tr
Fe	tr	¼	½¼	¼
Mn	tr	tr	¼	tr
Co	tr	tr	tr	tr
Ni	--	--	--	--

Conclusions:

1. The manganese minerals are relatively free from impurities.
2. The presence of barium reafirmed the identification of psilomelane.
3. The presence of manganese in the carbonate sample seemed to indicate a small amount of dolomite, $(Ca, Mg, Fe, Mn)CO_3$

Float-and-Sink Analysis

Float-and-sink tests are particularly valuable in ascertaining the distribution of the valuable minerals in various size ranges, in determining the extent of interlocking of minerals, and in preparing suitable portions for microscopic study.[1]

Acetylene tetbrabromide (sp. gr. 2.95) was used for all the float-and-sink tests, and carbon tetrachloride was used as the wash liquid.

The sized ore from the screening analysis was used for float and sink analysis. For particles coarser than 48 mesh the separation was effected in a 250 ml. beaker nearly filled with the heavy liquid. The minerals with a greater density than the fractionating medium sink while those with a lower density float. The float fraction was skimmed off with a small wire screen scoop, drained, and washed until all traces of the heavy liquid had disappeared. The product was then dried and weighed. The heavy liquid was then drained from the sink fraction. This product was also washed, dried, and weighed.

The technique for samples of finer size is similar to the above. The screened product was split to weigh from 15 to 25 grams. Separation was effected in a 300 ml. separatory funnel filled with the heavy liquid. The sink product was drawn off first and collected on a filter paper. The float product was then run onto a filter paper and both products washed, dried, and weighed.

Care must be taken in these tests to avoid mechanical entanglement of the sink products in the float and of the float in the sink. Shaking or stirring while the ore is still in the process of separating is usually sufficient to prevent this. The float-and-sink results are presented in Table IV.

1 - Report of Investigation 3328 U.S. Bureau of Mines

Table IV

Float and Sink Analysis

Mesh	Wt.%	Weight % Float	Weight % Sink	Assay % Float Mn	Assay % Sink Mn	Assay % Sink Fe	Assay % Insol.
-8+10	16.5	13.2	86.8	1.0	35.2	20.6	---
14	16.8	15.4	84.6	1.0	35.1	21.9	2.4
20	16.1	16.7	83.3	1.1	35.3	19.8	4.0
28	9.3	18.8	81.2	1.0	36.7	21.3	3.4
35	8.9	23.0	77.0	3.1	38.0	23.3	3.8
48	7.0	24.6	75.4	1.0	38.2	23.2	4.2
65	5.3	29.7	70.3	1.3	38.8	20.1	4.0
100	5.1	36.0	64.0	1.2	37.4	21.4	4.8
200	6.8	49.9	50.1	2.0	39.8	20.0	4.9
-200	8.2						

Table V shows the calculated assay percent of manganese in each screen size, and also the percent recovery of manganese in the sink products of each screen size.

Table V

Mesh	Product		Wt.%	Assay % Mn	Assay % Fe	Assay % Insol.	% Total Mn
-8+10	Float	2.95	13.2	1.0	----	---	0.4
	Sink	2.95	86.8	35.2	20.6	---	99.6
			100.0	30.66			100.0
14	Float	2.95	15.4	1.0	----	---	0.5
	Sink	2.95	84.6	35.1	21.9	2.4	99.5
			100.0	29.85			100.0
20	Float	2.95	16.7	1.1	----	---	0.6
	Sink	2.95	83.3	35.3	19.8	4.0	99.4
			100.0	29.58			100.0
28	Float	2.95	18.8	1.0	----	---	0.6
	Sink	2.95	81.2	36.7	21.3	3.4	99.4
			100.0	29.99			100.0
35	Float	2.95	23.0	3.1	----	---	2.4
	Sink	2.95	77.0	38.0	23.3	3.8	97.6
			100.0	29.97			100.0
48	Float	2.95	24.6	1.0	----	---	0.8
	Sink	2.95	75.4	38.2	23.2	4.2	99.2
			100.0	29.05			100.0
65	Float	2.95	29.7	1.3	----	---	1.4
	Sink	2.95	70.3	38.8	20.1	5.0	98.6
			100.0	27.69			100.0
100	Float	2.95	36.0	1.2	----	---	1.8
	Sink	2.95	64.0	37.4	21.4	4.8	98.2
			100.0	24.38			100.0
200	Float	2.95	49.9	2.0	----	---	4.8
	Sink	2.95	50.1	39.8	20.0	4.9	95.2
			100.0	20.94			100.0

Analysis of Sink Products

Briquettes of minus 8 plus 10, minus 28 plus 35, and 100 plus 200 mesh were now made of the sink products. These briquettes were made of lucite, as this material wets the mineral more than bakelite and prevents spalling of the specimens. An equal amount of ore and luxite was first mixed very thoroughly. This mixture was placed on the plunger of the briquetting machine, and a calculated amount of lucite added so as to give a briquette of approximately 15 cc. A pressure of 6000 pounds per square inch was applied to the plunger, and the mold was then heated to 85° C. At this time additional pressure was applied until there was a total pressure of 10000 pounds per square inch, and the heat was turned off. The temperature was allowed to drop to 50° and the briquette removed from the machine.

The briquette was first ground on a glass plate with 600 carborundum until the grains were exposed. The briquette was then given a more complete finish on the polishing machine using copper and lead laps, and finished on the tin oxide impregnated chamois lap.[1]

A study of the three briquettes seemed to indicate that the iron replaced the manganese, so at this time two specimens were removed from the minus 8 plus 10 mesh for spectrographic analysis. The spectrum showed one sample to contain much manganese and a trace of iron. The other sample contained a large amount of iron with a trace of iron. There was an insufficient amount of the minor element to uphold the indication of replacement between the iron and manganese minerals.

1 - Detailed explanation on Pp. 4.

Results of Float and Sink

Conclusions drawn from the float and sink tests are that the iron, manganese, and silicate minerals appear in the sink product. The assays show the manganese minerals to be the hardest as the total manganese is greater in the larger sizes. Although concentration was increased only from 27.2 per cent to 39.8 per cent manganese, tabling should be very advantageously carried out insofar as removal of the carbonate gangue is concerned.

Classification and Tabling

Should a preliminary examination of an ore indicate that gravity concentration is possible, a laboratory classification and tabling test is then made to ascertain the approximate per cent of recovery, the grade of concentrates obtainable, and the optimum degree of grinding.[1]

Classification and tabling was conducted as follows: A hindered-settling constriction-plate classifier was used for classification. The classifier tube is a pyrex glass 6" in diameter and 30" long. The constriction plate is a brass disk drilled with holes spaced at the vertices of equilateral triagles. A laboratory sized Wilfley table with a slime deck was used in tabling. The water feed and slope were adjusted for each spigot product.

A small amount of water was fed into the tube classifier while the ore was being charged. After charging, the hydraulic water was adjusted to give a slowly rising column of water to desliming the ore. After desliming the flow of water was increased until the ore was in a state of "tetter".

1 - U. S. Bureau of Mines R. I. 3328, Pp. 13

The ore was stirred occasionally to free any mechanically entrapped particles. The hydraulic water was then turned off and the ore allowed to settle. The number of spigot products into which the column was divided was determined by visual inspection. These were then syphoned off from the top downward and the products dried and weighed preparatory to tabling. In this test four spigot products, including the sands overflow product, were drawn off. These products were then tabled giving a concentrate, middling, and tailing product for each spigot product. The table products were then dried, weighed, and assayed. The classification and tabling results are presented in Table VI.

Table VI

Spigot Product No. 1 (coarse)

		Assay %			% Total
Table Products	Wt. (grs.)	Wt. %	% Mn	% Fe	Mn
Concentrates	2202	54.6	40.2	20.4	60.6
Middlings	1012	25.0	39.3	17.5	27.3
Tailings	825	20.4	21.5	14.2	12.1
	4039	100.0	36.2		100.0

Spigot Product No. 2

		Assay %			% Total
Table Products	Wt. (grs.)	Wt. %	% Mn	% Fe	Mn
Concentrates	1259	39.0	40.3	23.0	56.2
Middlings	857	26.6	31.7	19.9	30.1
Tailings	1110	34.4	11.2	10.8	13.7
	3226	100.0	29.2		100.0

Spigot Product No. 3

		Assay %			% Total
Table Products	Wt. (grs.)	Wt. %	% Mn	% Fe	Mn
Concentrates	564	19.1	37.0	25.7	30.5
Middlings	563	19.1	32.3	17.4	26.5
Tailings	1820	61.8	16.2	11.2	40.8
	2947	100.0	20.2		100.0

Spigot Product No. 4 (fine)

		Assay %			% Total
Table Products	Wt. (grs.)	Wt. %	% Mn	% Fe	Mn
Concentrates	251	20.1	35.5	23.8	31.6
Middlings	392	31.4	29.4	12.5	40.8
Tailings	605	48.5	12.9	11.3	27.6
	1248	100.0	22.6		100.0

Tabling proved very satisfactory. Number 1 and 2 products represent 65 per cent of the sample in weight and a recovery of approximately 85 per cent manganese. These products range in size from minus 8 to plus 35 mesh. Crushing should be such that most of the material is between minus 8 and plus 65 mesh, the range between which tabling is most effecient, and with improvement in the tabling operation their is no doubt of a higher recovery in all four products. As the slimes consist mostly of the softer carbonate minerals, there was very little loss of manganese in this product.

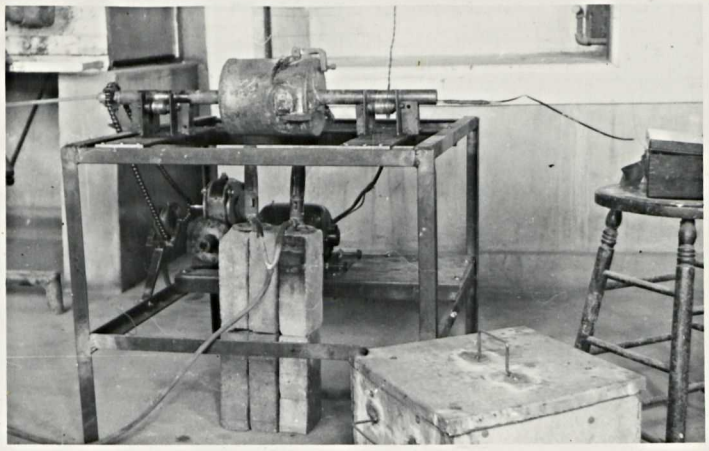
Roasting and Magnetic Separation

The table concentrates were now roasted in preparation for magnetic separation. Roasting was carried out in a reducing atmosphere. The roaster consisted of a revolving drum driven by a ¼ H. P. motor. A reducer was neccessary to regulate the speed of the drum, which was heated by a Bunsen burner, burning a mixture of gas and air under pressure. The reducing atmosphere was obtained by passing gas into the drum through one end of the hollow drive shaft and burning it at the other end. Plate III is a photograph of the roaster connected up and ready for operation.

Three roasts were carried out at this time. Equal proportions of No. 1, 2, and 3, table concentrates were mixed and thoroughly rolled. This mixture was split into three-500 gram samples. The first sample passed 8 mesh. The second sample was crushed on the bucking board to pass 35 mesh, and the third was crushed to pass 65 mesh. Roasting was conducted at a temperature of 450° C for 1-1/4 hours plus ½ hour required to heat the

ore to this temperature. The reducing atmosphere was retained until the roast had been quenched in a pan of water.

Plate III



The following method was used in calibrating the iron-capatan thermocouple used in checking roast temperature. A crucible of metal was heated until it became molten by means of a Bunsen burner. The thermocouple was placed in the molten metal and a reading taken on the millivoltmeter every half minute as the metal cooled. Cooling curves of lead, tin, aluminum, and zinc were charted in this way. The thermocouple was calibrated by plotting the known melting points of the metals against the meter reading. Plates IV and V show the cooling curve and the calibration curve, respectively.

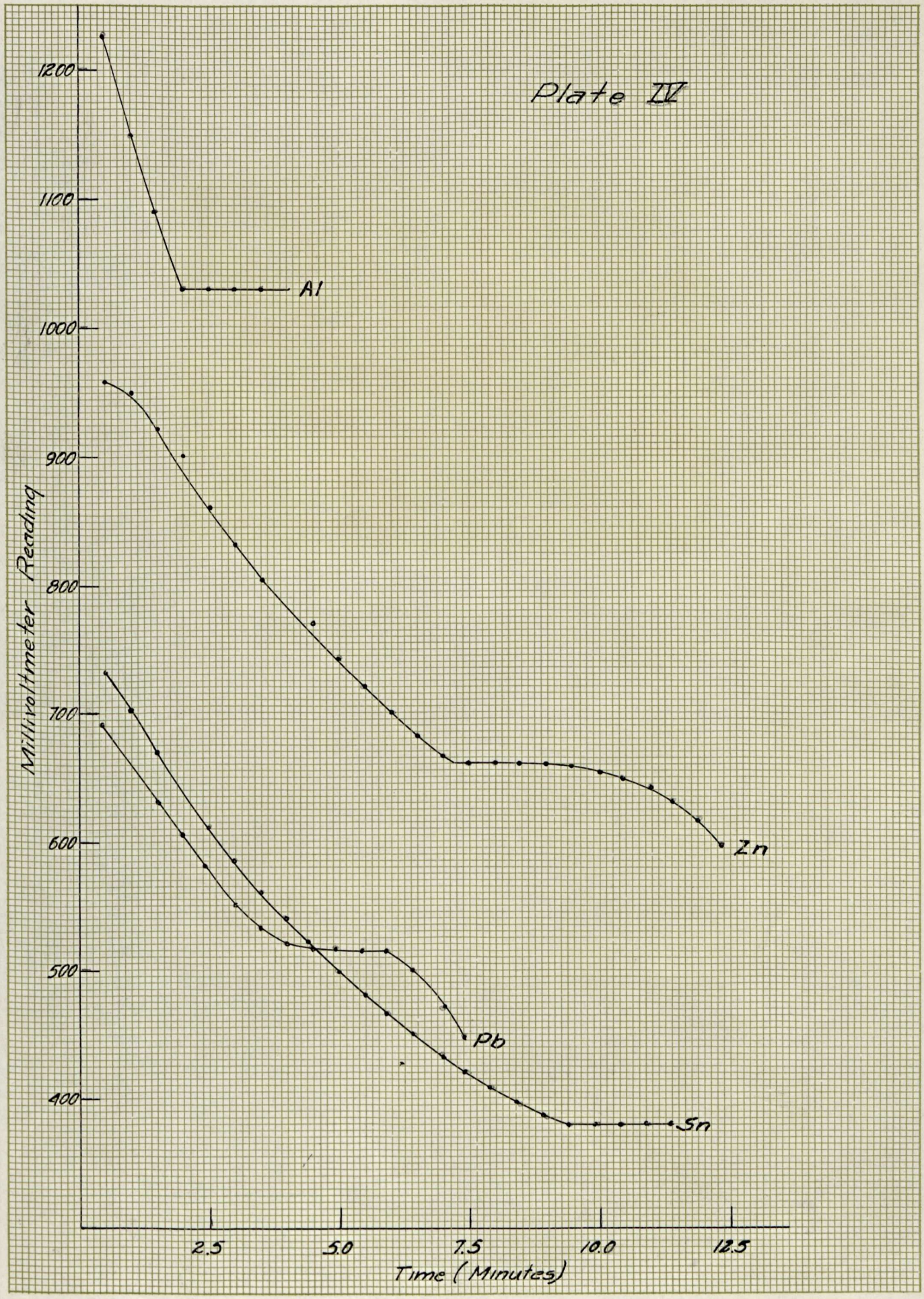
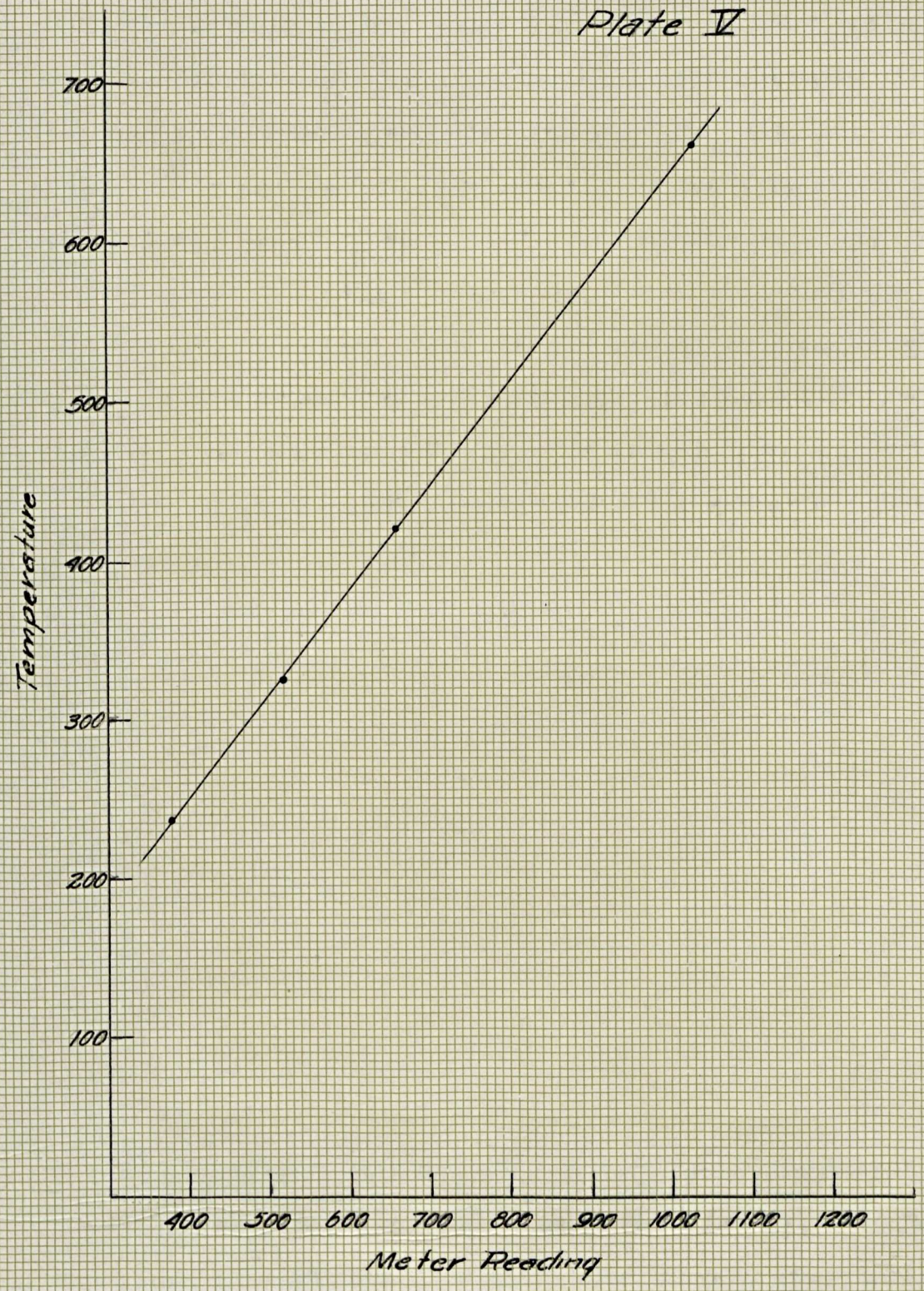


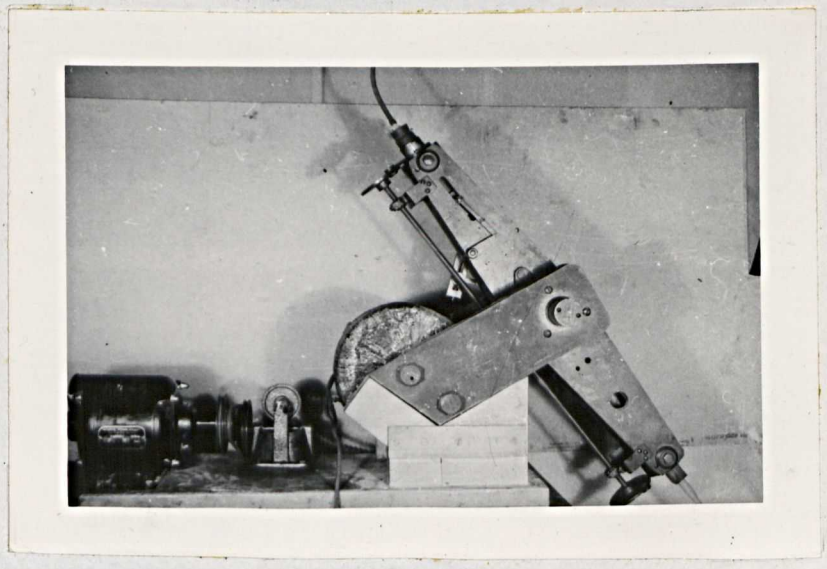
Plate IV
1200
1100
1000
900
800
700
600
500
400
Millivoltmeter Reading
Al
Zn
Pb
Sn
2.5
5.0
7.5
10.0
12.5
Time (Minutes)

Plate V



Magnetic separation was effected with the Davis Magnetic Tube Concentrator. The glass tube has a two way motion, a rotating motion and a shaking motion. The magnetic field was induced by a motor- generator set-up. The magnetic material was held in suspension about midway up the tub, while the rest of the material sank to the bottom where it was drained into a pan. A steady stream of water was fed into the tube as draining proceeded which maintained a constant level of water in the tube. When the non-magnetic material had been drained, the magnetic material was washed into another pan. Plate Vi is a photograph of the magnetic separator used in this work.

Plate VI



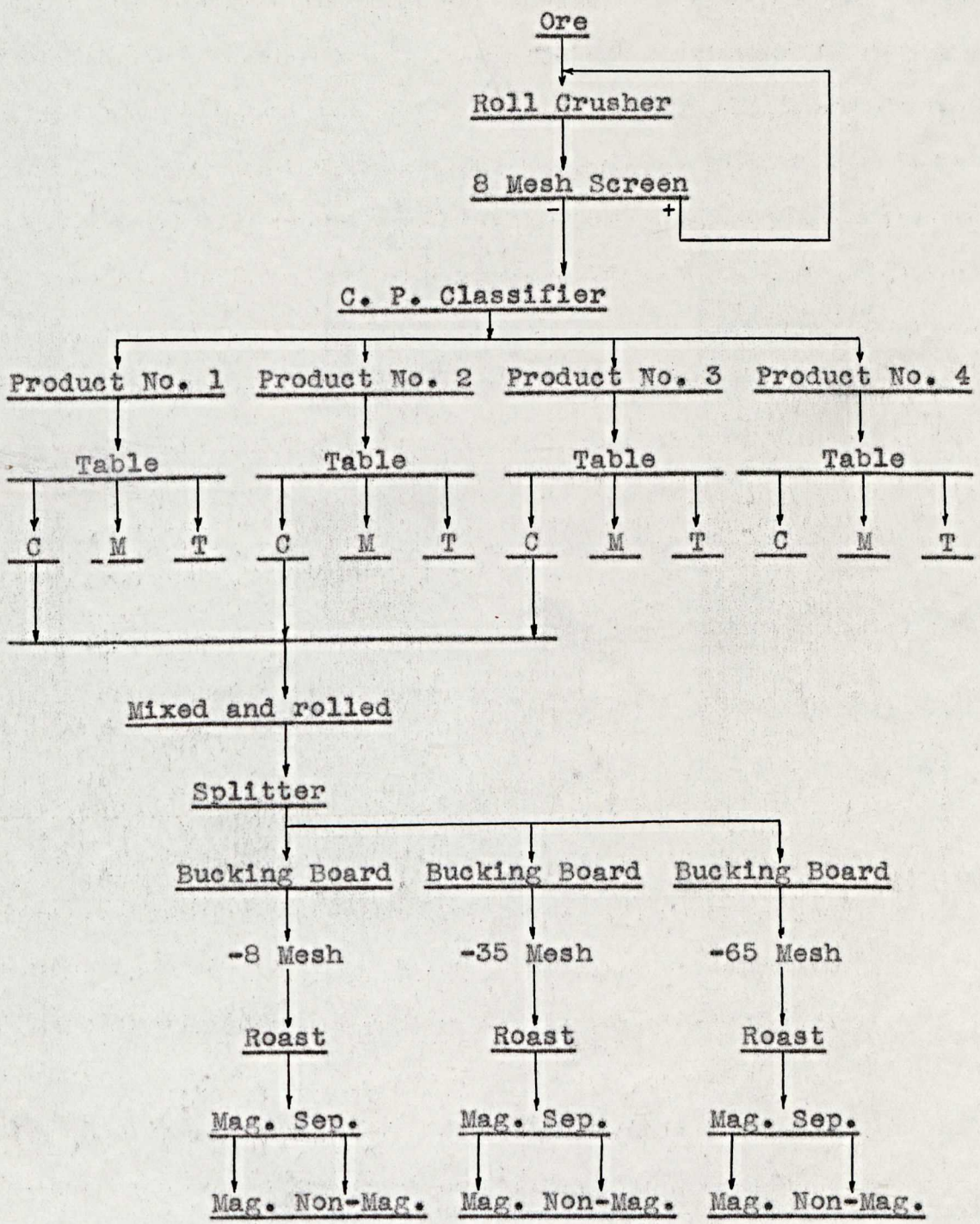
The products of No. 1, 2, and 3, roasts were split to approximately 25 grams, and a magnetic separation effected. The results were very poor with only a small amount of magnetic material present.

The reason for the small amount of material in these first three roasts was thought to be that air was being drawn into the drum along with the gas.

Two more 500 gram samples of the table concentrates were roasted at this time. One sample passed 8 mesh and the other was ground to pass 35 mesh. The temperature was held at 500° C for two hours. The 8 mesh roast was quenched in water, while the 35 mesh roast was cooled under a reducing atmosphere. The shaft at the gas entrance, was fixed so that no air could be drawn into the drum.

These samples were split to approximately 25 grams and a magnetic separation effected. There was a greater amount of magnetic material in the 8 mesh roast than in the 35 mesh roast although there was no more than 5% magnetic material in this sample.

Laboratory Flowsheet



Conclusions and Discussion for Further Study

It is believed that this ore can be effectively concentrated for shipment, although the economical aspect is doubtful because of the low grade of the deposits. Classification and tabling can be used to a great advantage for the removal of the carbonate material producing a concentrate containing 40% man ganese.

Roasting was not successful. On the last two roasts the reduced ore was reoxidized by some unknown mechanism. The temperature at which the last two roasts were carried out should be sufficient for complete reduction of hematite to magnetite. The time of roasting may have to be lengthened. Quenching in water seemed to produce better results than cooling in a reducing atmosphere.

Further work would be experimentation of roasting procedure with the re-oxidation of the reduced material being the main difficulty which must be eliminated.

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Acknowledgement

The writer expresses his appreciation to Dr. S. R. B. Cooke, head of the Department of Mineral Dressing for his assistance in the work represented by this thesis.

The writer is also indebted to the graduate students: Messrs. Jack L. Taylor, Robert Wilson, and Carlos Plenge for their help and suggestions.